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**(54) Title of the Invention:** Resin Composition for Powder Coating

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**SPECIFICATION**

**1. Title of the Invention**

Resin Composition for Powder Coating

**2. Claims**

A resin composition for a powder coating consisting of (1) 50-95 wt% of a polyester resin with a hydroxyl value of 10-100 KOH mg/g, an acid value of 20 KOH mg/g or less, and a number average molecular weight of 1000-10,000; (2) 0.5-30 wt% of a polyester resin with an acid value of 30-300 KOH mg/g, number average molecular weight of 500-10,000, and an average of two or more carboxyl groups per molecule, (3) 3-40 wt% of blocked polyisocyanate, and (4) 0.5-20 wt% of an epoxy resin.

### 3. Detailed Description of the Invention

The present invention relates to a resin composition for powder coating that has a good ability to cover the edge parts together with excellent coating film formation.

Polyester resin powder coatings that employ blocked polyisocyanate as the curing agent are inferior in their ability to cover the edge parts, as is commonly known. The use of compounds to impart thixotropy or large amounts of filler has been considered as means for resolving this, but not only is the edge covering ability inadequate, another drawback is that the inherently excellent coating film-forming ability of the polyester resin powder coating deteriorates. Priming with a rust-proofing paint has been done in the past in an attempt to compensate for the poor edge covering ability, but necessitates a complicated operation.

The present invention improves upon these drawbacks and relates to a resin composition for powder coating consisting of (1) 50-95 wt% (shown by % hereinafter) of a polyester resin having an abundance of hydroxyl groups, (2) 0.5-30% of a polyester resin with an abundance of carbonyl groups, (3) 3-40% of blocked polyisocyanate, and (4) 0.5-20% of an epoxy resin.

The polyester resin with a high hydroxyl value used in the present invention is the main component that reacts with the blocked polyisocyanate. The hydroxyl value of this polyester resin is 10-100 KOH mg/g (the unit is omitted hereinafter), and preferably 20-80. The acid value is 20 KOH mg/g (the unit is omitted hereinafter) or less, and preferably 15 or less. Its number average molecular weight is 1000-10,000, and preferably 2000-6000. The softening point, together with the polyester resin discussed below, is 80-130°C, and preferably 90-120°C.

The polyester resin with many carboxyl groups has an average of two or more, and preferably three or more, carboxyl groups per molecule. It reacts mainly with the epoxy resin, but if hydroxyl groups are present, it also reacts with the dissociated isocyanate groups of the blocked polyisocyanate. The acid value of this polyester resin is 30-300, and preferably 50-250. The hydroxyl value is not particularly restricted. There need be almost no hydroxyl groups, but at most the hydroxyl value should be 30 or less, not to exceed half the total hydroxyl value of the polyester resin. Its number average molecular weight is 500-10,000, and preferably 1000-6000.

These polyester resins can be obtained by reacting a carboxylic acid and an alcohol by a commonly known method. Examples of carboxylic acids that can be used include terephthalic acid, isophthalic acid, phthalic acid, lower alkyl esters of these acids, trimellitic acid, pyromellitic acid, adipic acid, suberic acid, sebacic acid, dodecanoic diacid, dodecynyl succinic anhydride, dimer acid, and tetrahydrophthalic acid. Examples of alcohols include ethylene glycol, propylene glycol, neopentyl glycol, 1,6-hexanediol, trimethylolethane, trimethylolpropane, pentaerythritol, hydrogenated bisphenol A, and bis-2-hydroxyethyl terephthalate. A portion of these carboxylic acids or alcohols can also be replaced by an oxy acid such as 12-hydroxybenzoic acid.

The blocked polyisocyanate reacts with the hydroxyl groups in the polyester resin by producing free isocyanate groups by block dissociation. It also reacts with hydroxyl groups produced by reactions between the carboxyl groups of the polyester resin and the epoxy groups of the epoxy resin. The blocked isocyanate here is one in which the free isocyanate groups have been blocked by reacting a blocking agent with a polyisocyanate or an adduct of a polyisocyanate and polyhydric alcohol. Examples of the polyisocyanate include isophorone diisocyanate, tetramethylene diisocyanate, hexamethylene diisocyanate, lysine diisocyanate, and xylylene diisocyanate. Examples of the polyhydric alcohol include trimethylolpropane, glycerin, and ethylene glycol. Examples of the blocking agent include methanol, ethanol,  $\epsilon$ -caprolactam, 2-pyrrolidone, acetoxime, and phenol. The amount of blocked polyisocyanate used is 3-40% as was already stated, but a range of 5-30% is more preferred.

The epoxy resin is the component that reacts with the carboxyl groups in the polyester resin. It has an average of two or more epoxy groups per molecule. Examples of such epoxy resins include triglycidyl isocyanate, bisphenol-type epoxy resin, hydrogenated bisphenol-type epoxy resin, and ester-type epoxy resin. The amount of epoxy resin used is 0.5-20% as was already stated, but a range of 1-10% is more preferred.

The composition of the present invention is made into a powder coating by combining pigments and other fillers, fluidity adjusters, and sometimes catalysts and other additives with it and kneading and then pulverizing finely by an extruder. Examples of catalysts that can be used include organic tin compounds and tertiary amine compounds. Examples of fluidity adjusters and

other additives include acrylic polymers, silicone compounds, and fluorine compounds. The powder coating obtained is applied to a base material, and a cured coating film with excellent properties is obtained by baking at 150°C or higher, and preferably 160-220°C.

The present invention is explained below through working examples. "Parts" in the text refers to parts by weight.

#### Synthesis of polyester resin I

Twenty-four parts of propylene glycol, 120 parts of 1,6-hexanediol, 855 parts of neopentyl glycol, 72 parts of bis-2-hydroxyethyl terephthalate, 926 parts of dimethyl terephthalate, and 0.7 part of zinc acetate were heated. After heating gradually from 160 to 220°C while removing the methanol, 790 parts of isophthalic acid and 89 parts of trimethylolpropane were added, and a polyester resin I with a hydroxyl value of 68, acid value of 8, softening point of 104°C, and number average molecular weight of 1800 was obtained by heating for 12 hours at 240°C.

#### Synthesis of polyester resin II

A polyester resin II with a hydroxyl value of 34, acid value of 6, softening point of 115°C, and number average molecular weight of 4400 was obtained in the same way as in the synthesis of polyester resin I using 218 parts of ethylene glycol, 158 parts of neopentyl glycol, 467 parts of dimethyl terephthalate, 21 parts of trimethylolpropane, and 393 parts of terephthalic acid.

#### Synthesis of polyester resin III

One hundred fourteen parts of neopentyl glycol, 1114 parts of bis-2-hydroxyethyl terephthalate, 650 parts of isophthalic acid, and 1.5 parts of dibutyl tin oxide were heated. After gradually heating from 160 to 220°C while removing the methanol, 100 parts of trimellitic anhydride were added, and a polyester resin III with a number average molecular weight of 1500, softening point of 94°C, acid value of 151, hydroxyl value of zero, and an average of four carboxyl groups per molecule was obtained by holding for one hour at the same temperature.

## Synthesis of polyester resin IV

An amount of 297 parts of propylene glycol, 242 parts of ethylene glycol, 1364 parts of dimethyl terephthalate, and 1 part of zinc acetate was heated. After heating gradually from 160 to 210°C while removing the methanol, 210 parts of trimellitic anhydride were added, and a polyester resin IV with a number average molecular weight of 2400, softening point of 90°C, acid value of 82, hydroxyl value of 12, and an average of 3.5 carboxyl groups per molecule was obtained by holding for one hour at the same temperature.

## Working Example

After dry blending the components shown in Table 1 (the amounts used are shown by parts), a white paint was obtained by passing the mixture through a 200 mesh metal screen while kneading by a PR46 cokneader made by Booth. This powder coating was applied to a zinc phosphate-treated iron plate (thickness 2 mm, length 150 mm, width 70 mm) by electrostatic spraying and baked for 20 minutes at 200°C. The coating film performance of the approximately 60 micron-thick film was as shown in Table 1.

Table 1

	Working Example 1	Working Example 2	Working Example 3
Polyester resin I	70		
Polyester resin II		85	85
Polyester resin III	5		5
Polyester resin IV		4	
Crelan UI <sup>1)</sup>	30		15
Adact B1065 <sup>2)</sup>		15	
Triglycidyl isocyanate	2		2
Epiclon 4050 <sup>3)</sup>		6	
Modaflow <sup>4)</sup>	1	1	1
Titanium oxide	43	43	43
Smoothness	Good	Good	Good
60° reflected light gloss	98	98	96
Eriksen (mm)	>7	>7	>7
Impact resistance (500 g× cm)	30	50	50
Tight adhesiveness <sup>5)</sup>	100/100	100/100	100/100
Edge covering (visual)	Good	Very good	Very good
Moisture resistance: rust after 200 hours <sup>6)</sup>	No	No	No
Rust after saline spray <sup>7)</sup>	No	No	No

1) Made by Bayer, ε-caprolactam-blocked isophorone diisocyanate adduct, blocked isocyanate group content 11.5%.

2) Made by Feba-Hemi [phonetic], blocked isophorone diisocyanate adduct, blocked isocyanate group content 10.5%.

3) Made by Dainippon Ink and Chemicals, bisphenol-type diepoxide, number average molecular weight approximately 2000.

4) Made by Monsanto, acrylic polymer fluidity adjuster.

5) Number of squares left when 11 cuts were made lengthwise and widthwise in a 1 mm area, cellophane tape was affixed and then pulled away.

6) Extent of rust development from the edges under 98% humidity at 50°C.

7) Extent of rust development after spraying with 35°C, 5% saline.